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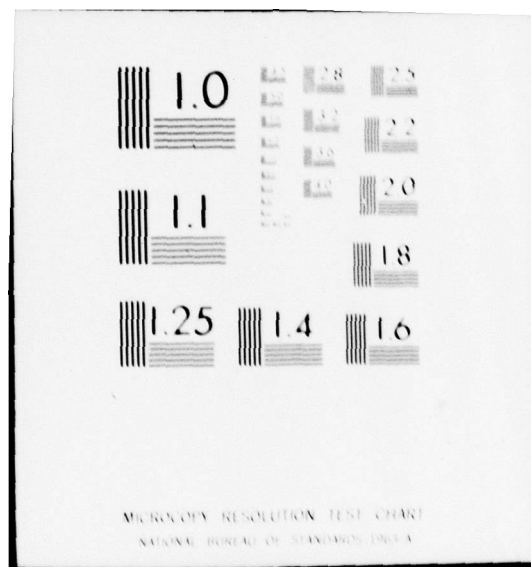
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# AERONAUTICAL ANALYTICAL REWORK PROGRAM

## FINAL REPORT

### FIELD DETERMINATION OF THE ACIDITY OF MIL-L-23699 LUBRICATING OIL

FEBRUARY 1978

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Final Report  
Modify Oil Contamination Kit  
62269/TR7-1573  
77 June 02 to 77 December 29

6 FIELD DETERMINATION OF THE ACIDITY  
OF MIL-L-23699 LUBRICATING OIL.

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ABSTRACT

Constant speed drive failures associated with the MIL-L-23699 oil degradation required a feasibility study to determine techniques/procedures satisfactory for the field evaluation of oil samples. Results of the study showed that a test procedure based on total acid number was capable of discriminating between MIL-L-23699 oils with total acid numbers above and below 2.0 mg KOH/gram. A prototype test kit (go-no go type) is being assembled for on-site field demonstrations. This maintenance technology support study was conducted under the Analytical Rework Program sponsored by the Naval Air Systems Command (AIR-4114C).

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## REPORT OF INVESTIGATION

### 1. Introduction

The purpose of this investigation has been to develop a procedure for the field evaluation of the acidity of MIL-L-23699 lubricating oils. The requirements for the procedures to be developed (Reference 1) are that they shall be capable of distinguishing, on a "go-no go" basis, between oils of acidity greater or lesser than 2.0 mg.KOH per gram; that they shall be field tests which can be performed by non-technically trained personnel; and that they shall be applicable to MIL-H-23699 lubricant oils which contain either additive group A or additive group B.

A field test procedure has been developed at this laboratory for the field evaluation of compressor lubricant acidity. (References 2 and 3). That procedure, which is applicable to additive-free mineral oils, is capable of distinguishing between used oils which have acidities greater or less than 0.05 mg.KOH/gram. The principle of this field test for compressor lubricant acidity is the extraction of acidic components formed within the used oil into a solvent which is both immiscible with the compressor oil sample and capable of dissolving the acid components of the sample with a high degree of efficiency. It was felt, a priori, that the principle of the compressor lubricant test kit might be applied to MIL-L-23699 oils. Therefore, the initial studies in the present investigation were directed toward that end.

It was found, in fact, that a very satisfactory test could be developed in the above manner for the evaluation of acidity at the 2.0 mg. KOH/gram level in MIL-L-23699 oil with additive group A. The same test, however, could not be used for MIL-L-23699 oils containing additive group B because that additive package developed a strong purple color in alkaline solutions. That color interfered with the evaluation of the test results by the procedure described above. The bulk of the research subsequently performed in this program was directed toward the elimination of additive interference in oils containing the group B additive package. Several different approaches were investigated. These included the evaluation of a variety of solvent systems, the development of emulsion tests and of procedures with indicating upper phases instead of the indicating lower phases as employed in the compressor lubricant test procedure.

In the work to be described in subsequent sections of this report several experimental procedures have been developed. These were tested initially by application to new MIL-L-23699 oils of both types to which known amounts of oleic acid had been added. Final evaluation of the efficacy of prospective test procedures was made by use of actual used oils obtained from field applications.



## 2. Samples

The following samples have been utilized throughout the course of this investigation.

Laboratory Number	Sample Of	Marked	Total Acid No., mg.KOH/gram (ASTM D 664)
7 7 18 3	New MIL-L-23699 Oil	Additive group A	0.17
7 7 18 4	New MIL-L-23699 Oil	Additive group B	0.17
7 7 7 10	New MIL-L-23699 Oil	Additive group B, 0.10 TAN	0.28
7 7 7 11	Used MIL-L-23699 Oil	Additive group B, 2.52 TAN	2.68
7 7 7 12	Used MIL-L-23699 Oil	Additive group A, 2.60 TAN	2.86
7 7 7 13	Used MIL-L-23699 Oil	Additive group B, 5.23 TAN	5.67
7 7 7 14	Used MIL-L-23699 Oil	Additive group B, 3.15 TAN	3.47
7 7 7 15	Used MIL-L-23699 Oil	Additive group A, 3.25 TAN	3.17
7 7 7 16	Used MIL-L-23699 Oil	Additive group B, 6.12 TAN	6.06
7 7 7 17	Used MIL-L-23699 Oil	Additive group A 4.20 TAN	4.14
7 7 7 18	Used MIL-L-23699 Oil	Additive group B, 1.66 TAN, Hydrolysis possible	2.25
7 7 7 19	Used MIL-L-23699 Oil	Additive group B, 0.43 TAN, Hydrolysis possible	1.42
7 7 7 20	Used MIL-L-23699 Oil	Additive group B, 1.58 TAN, Hydrolysis possible	2.44
7 7 7 21	Used MIL-L-23699 Oil	Additive group B 0.75 TAN, Hydrolysis possible	1.03

The acidities (TAN) supplied as part of the markings of the samples as received from the Department of the Navy, Naval Air Propulsion Test Center, Trenton, NJ 08628, were found to differ somewhat from the values determined in this laboratory as indicated above. This result was expected since experience had shown that the samples were unstable with respect to their acidity (Reference 4). The measured acidities did not, however, change during the time the samples were at this laboratory and the values recorded above were used for all test purposes.

### 3. Experimental

- 3.1 The following data were obtained during the course of this investigation. The reagents used in each phase are described in the appropriate sections. All reagent and sample volumes were measured with standard volumetric glassware into mixing bottles. The mixing bottles were one oz. clear glass, French Square bottles with screw caps.
- 3.2 New oil of different acidities was prepared by adding oleic acid to Lab. No. 7 7 18 3 (additive group A). The following acidities were prepared: 1.72, 1.81, 1.91, 2.01, 2.12 and 2.22 mg.KOH/gram. Then 5 ml of each oil sample were introduced into a shaking bottle; 5 drops of phenolphthalein indicator were added; and in the first case 10 ml of a solvent consisting of 50% v of toluene and 50% v isopropyl alcohol. Finally 10 ml of aqueous 0.0099 N sodium hydroxide were added and the mixture shaken. An additional 5 ml of 0.0099 N sodium hydroxide were then added and the mixture shaken again. In the second case the titration solvent consisted of pure toluene. The following observations were then made of the aqueous layer that separated on standing:

Total Acid No. of Sample mg.KOH/gram (Lab. No. 7 7 18 3)	Case I		Case II
	10 ml NaOH added	15 ml NaOH added	10 ml NaOH added
1.72	Colorless	Light pink	Red
1.81	Colorless	Light pink	Red
2.01	Colorless	Light pink	Red
2.12	Colorless	Light pink	Light pink
2.22	Colorless	Light pink	Light pink

These data show that the efficiency of extraction of acidic components by the aqueous caustic is dependent on the nature of the solvent used and the relative amounts of aqueous and non-aqueous phases. Further examination showed that the solvents were not neutral. This indicated that all solvent added should be adjusted to the pH of the change point of the indicator. The separation of layers was slow but seemed to be faster for the tests run with smaller amounts of the aqueous phase.

- 3.3 Titration solvent consisting of 50% v toluene, 49.5% v isopropyl alcohol and 0.5% v distilled water was adjusted to pH 10.3 with dilute aqueous sodium hydroxide. Then 5 ml of the solvent, 5 ml of Lab. No. 7 7 18 3 oil adjusted to a TAN of 2.01 mg.KOH/gram and 5 drops of phenolphthalein indicator were transferred to shaking bottles containing different amounts of 0.0297 N aqueous sodium hydroxide. The mixtures were then shaken and allowed to stand until they separated into an aqueous lower layer and an upper organic layer. The following observations were then made:

Lab. No. 7 7 18 3 Adjusted to TAN 2.01 mg.KOH/gram

Volume of 0.0297 N NaOH Added, ml.	Color of aqueous Lower layer
6.2	Colorless
6.3	Colorless
6.4	Very faint pink
6.5	Faint pink
6.6	Light pink

These data show that a reasonable change point can be obtained for TAN of 2.0 mg.KOH when about 6.4 ml of 0.0297 N NaOH is used with neutralized titration solvent of the above indicated type. This finding suggests an approximate 76% efficiency in the extraction of acidic components into the aqueous phase and led to the following experiment:

- 3.4 A measured quantity (5 ml) of the standard oil Lab. No. 7 7 18 3 adjusted to different acidities was added to each of several shaking bottles; 10 ml of neutralized titration solvent (see par. 3.3); 6.4 ml of 0.0297 N aqueous sodium hydroxide; and 5 drops of phenolphthalein indicator were added to each and the mixtures shaken and allowed to stand until separation occurred.



The following observations were then made:

Total Acid No. of Sample mg.KOH/gram (Lab. No. 7 7 18 3)	Color of Lower Layer
1.72	Pink
1.81	Pink
1.91	Light pink
2.01	Faint pink
2.12	Colorless
2.22	Colorless

The separation time of the layers was less than two minutes in each case. On the basis of the data obtained it was concluded that the described procedure was capable of testing the acidity of MIL-L-23699 oil with the group A additive package on a "go-no go" basis with a change point at 2.0 mg.KOH/gram. The next step was to try the same procedure on oil containing additive group B (Lab. No. 7 7 18 4).

- 3.5 Standards of different acidity were prepared by the addition of oleic acid to Lab. No. 7 7 18 4 which was a MIL-L-23699 oil containing additive group B. These were then tested in accordance with the procedure described in par. 3.4. The following results were obtained.

Total Acid No. of Sample mg.KOH/gram (Lab. No. 7 7 18 4)	Color of Lower Layer
1.59	Pink
1.81	Pink
1.89	Pink
1.98	Pink
2.06	Light pink
2.22	Light pink

No clear cut color difference was observed, as expected, between samples with acidities greater or less than 2.0 mg.KOH/gram. Moreover, the quality of the color was different from that observed in the previous test. This suggested that an interference might be present. This was evaluated in the following manner:

- 3.6 Standard oil (Lab. No. 7 7 18 4) adjusted to a TAN of 1.98 mg.KOH/gram was prepared and 5 ml were added to each of 12 shaking bottles. Then 10 ml of neutralized titration solvent (see par. 3.3) and various amounts of 0.0297 N aqueous sodium hydroxide were added. No indicator was added. The mixtures were shaken, allowed to stand until separation occurred and the following observations were made:

Lab. No. 7 7 18 4 Adjust to TAN 1.98 mg.KOH/gram

Volume of 0.0297 N. NaOH added, ml.	Color of Lower Layer
6.4	Pink
6.1	Pink
5.9	Pink
5.7	Pink
5.5	Pink
5.3	Pink
5.1	Light pink
4.9	Light pink
4.7	Light pink
4.5	Light pink
4.3	Light pink
4.1	Light pink

It was further observed that, if the above mixtures were shaken a second time, they did not separate upon standing. However, a separated layer could be obtained by the centrifuging of the samples. In that case the lower layer was reproduced without substantial change in color. The same series of experiments were repeated without substantial change in result for a sample of standard oil (Lab. No. 7 7 18 4) adjusted to a TAN of 2.22 mg.KOH/gram.



From these findings it is clear that the oil sample containing the group B additive package transmits a color to the separated aqueous layer. This color is only slightly dependent on the amount of excess base and for this reason constitutes a serious interference to the test procedure for the MIL-L-23699 oil with the type B additive package. Further test development was, therefore, continued as follows:

- 3.7 It was determined (Reference 5) that the component of additive group B that was most likely to produce this anomalous color change was 1,4-dihydroxyanthraquinone. To test this hypothesis the following tests were made: Oil containing the type A additive package (Lab. No. 7 7 18 3) was adjusted to various total acidities with oleic acid. Then 5 ml each of various oils, 10 ml of neutralized titration solvent (see par. 3.3), 6.8 ml of 0.0297 N aqueous sodium hydroxide, 3 ml of 0.5% w 1,4-dihydroxyanthraquinone in titration solvent, and 5 drops of phenolphthalein solution were placed in shaking bottles, mixed and allowed to stand. The following observations were made:

Total Acid No of Sample mg.KOH/gram (Lab. No. 7 7 18 3)	Color of Emulsion Before Separation	Color of Separated Lower Layer
1.72	Rust	Dark Pink
1.81	Light Rust	Pink
1.91	Dark Yellow	Pink
2.01	Dark Yellow	Pink
2.12	Dark Yellow	Pink
2.22	Dark Yellow	Pink

These findings confirm that 1,4-dihydroxyanthraquinone is the source of the interference first observed in the testing of oils containing the group B additive package by the method of par. 3.4. A further, and significant, finding is that the color of the emulsion formed before separation can be used as an indication of sample acidity.

- 3.8 Standard samples of oil (5 ml) with the type B additive group (Lab. No. 7 7 18 4) with acidities adjusted by the addition of oleic acid were added to 10 ml of neutralized titration solvent (see par 3.3), 5 drops phenolphthalein solution, and 6.4 ml of aqueous 0.0297 N sodium hydroxide. The mixtures were shaken until and emulsion formed and the following observations were made:

Total Acid No. of Sample mg.KOH/gram (Lab. No. 7 7 18 4)	Color of Emulsion Before Separation (6.4 ml aqueous NaOH solution)
---	--

1.59	Purple-Red
1.81	Red-Brown
1.89	Rust
1.98	Light Rust
2.06	Yellow
2.22	Yellow

It was found that, although the color change observed for the oil containing the type B additive group was similar to that observed for the material containing the type A additive (see par. 3.7), the change point was different when identical amounts of reagent were used and the quality (hue) of the developed colors was different under otherwise identical circumstances. The effect was quantified as follows: to 5 ml of Lab. No. 7 7 18 3 adjusted to a TAN of 1.80 mg.KOH/gram were added 10 ml of neutralized titration solvent (see par. 3.3), 1.0 ml 0.5% w 1,5-dihydroxyanthraquinone solution, 5 drops of phenolphthalein indicator and varying amounts of aqueous 0.0297 N aqueous sodium hydroxide solution. It was found that 7.2 ml of the aqueous sodium hydroxide solution were required to develop an emulsion color identical to that obtained above under the indicated conditions for the Lab. No. 7 7 18 4 mixtures (par. 3.8). This finding led to the following experiments:

- 3.9 To 5 ml samples of Lab. No 7 7 18 3 (additive group A) adjusted to different acidities were added 10 ml of neutralized titration solvent (see par. 3.3), 1.0 ml of 0.5% w 1,4-dihydroxyanthraquinone solution, 5 drops of phenolphthalein indicator solution and 7.2 ml of 0.0297 N aqueous sodium hydroxide solution. The mixtures were shaken until emulsions formed and the following observations were made:

Total Acid No. of Sample mg.KOH/gram (Lab. No. 7 7 18 3)	Color of Emulsion Before Separation (7.2 ml NaOH Solution added)
---	--

1.72	Violet
1.81	Rust
1.91	Light Rust
2.01	Yellow
2.12	Yellow
2.22	Yellow

A further adjustment was made as follows to improve the correlation of change point colors with those obtained for Lab. No. 7 7 18 4 mixtures (par. 3.8).

Total Acid No. of Sample, mg.KOH/gram (Lab. No. 7 7 18 3)	Color of Emulsion Before Separation (7.5 ml NaOH Solution added)
1.72	Violet
1.81	Violet
1.91	Rust
2.01	Very light rust
2.12	Yellow
2.22	Yellow

The following experiment was then run with oils containing additive group B (Lab. No. 7 7 18 4): 5 ml of oil adjusted to the indicated TAN, 10 ml of neutralized titration solvent, 5 drops of phenolphthalein indicator and 6.5 ml of aqueous 0.0297 N sodium hydroxide solution were shaken until an emulsion formed. Note: No 1,4-dihydroxyanthraquinone solution was added. The following observations were then made:

Total Acid No. of Sample, mg.KOH/gram (Lab. No. 7 7 18 4)	Color of Emulsion Before Separation (6.5 ml of NaOH solution added)
1.59	Violet
1.81	Light Violet
1.89	Rust
1.98	Light Rust
2.06	Yellow
2.22	Yellow

3.10 It was clear that the data obtained from the experiments described in par. 3.9 could form the basis of an acid number test. Several experiments were therefore, conducted with used oil samples of known acid number. While the procedure was found to work satisfactorily for light colored used oils, dark colored samples interfered with the clear observation of the color of the emulsion formed upon shaking the samples with the indicated reagents. It was, therefore, found to be necessary to continue the test development with emphasis directed towards a procedure which resulted in the separation of an indicator-containing liquid phase. The following experiment was performed: 5 ml samples of Lab. No. 7 7 18 4 oil adjusted to different acidities were added to 100 ml of neutralized titration solvent, 20 drops of 0.5% w 1,4 dihydroxyanthraquinone, 20 drops of phenolphthalein solution and 6.4 ml of aqueous 0.0297 N sodium hydroxide solution and shaken. A clear solution resulted. Then 20 ml of distilled water were added and the mixture shaken again. Upon standing an aqueous lower layer separated. The following observations were made:

Total Acid No. of Sample, mg.KOH/gram (Lab. No. 7 7 18 4)	Color of Clear Solution	Color of Separated Lower Layer
1.59	Purple-red	Pink
1.81	Reddish Brown	Pink
1.89	Rust	Light Pink
1.98	Orange	Very Light pink
2.06	Orange	Colorless
2.22	Orange	Colorless

While the above procedure appeared to show promise as a test for the field evaluation of the acidity of MIL-L-23699 oils, it was felt that the solvent volumes involved were too large to be practical and that further development was required. This led to the following test procedure:

3.11 For MIL-L-23699 oils containing the type A additive package (Lab. No. 7 7 18 3) add 5 ml of oil to 20 ml of acetone, 1.0 ml of 0.5% w 1,4-dihydroxyanthraquinone solution, 10 drops phenolphthalein indicator solution, and 7.3 ml of aqueous 0.0297 N sodium hydroxide. Shake well and observe the color of the separated upper solvent layer. Thus:



Total Acid No. of Sample, mg.KOH/gram (Lab. No. 7 7 18 3)	Color of Separated Upper Layer (7.3 ml of NaOH solution added)
--	--

1.72	Dark Violet
1.81	Dark Violet
1.91	Reddish Brown
2.01	Dark Straw
2.12	Yellow
2.22	Yellow

For MIL-L-23699 oils containing the type B additive package (Lab. No. 7 7 18 4) add 5 ml of oil to 20 ml of acetone, 10 drops of phenolphthalein indicator solution and 6.3 ml of aqueous 0.0297 N sodium hydroxide solution. Shake well and observe the color of the separated upper solvent layer. Thus:

Total Acid No. of Sample, mg.KOH/gram (Lab. No. 7 7 18 4)	Color of Separated Upper Layer (6.3 ml of NaOH solution added)
--	--

1.59	Dark Violet
1.81	Dark Violet
1.89	Reddish Brown
1.98	Dark Straw
2.06	Yellow
2.22	Yellow

In both cases the time required for the required separation of layers to occur was less than three minutes. It was thus considered that the tests should be applied to the several samples of used oils. The following results were obtained:



## 3.12

Laboratory Number	Additive Type	Total Acid No., mg.KOH/gram (ASTM D 664)	Color of Separated Upper Layer	Test Indication
7 7 7 10	B	0.28	Dark Violet	Pass
7 7 7 11	B	2.68	Yellow	Fail
7 7 7 12	A	2.86	Dark Yellow	Fail
7 7 7 13	B	5.67	Yellow	Fail
7 7 7 14	B	3.47	Yellow	Fail
7 7 7 15	A	3.17	Dark Yellow	Fail
7 7 7 16	B	6.06	Yellow	Fail
7 7 7 17	A	4.14	Yellow	Fail
7 7 7 18	B	2.25	Dark Yellow	Fail
7 7 7 19	B	1.42	Dark Violet	Pass
7 7 7 20	B	2.44	Yellow	Fail
7 7 7 21	B	1.03	Dark Violet	Pass

A further observation was made: namely, that the color of the lower layer of the sample reagent mixture before shaking was, except in the case of extremely oxidized or hydrolyzed samples (cf Lab. Nos. 7 7 7 16 and 7 7 7 18) violet for oils containing the type B additive package and colorless for those containing the type A additive package.

#### 4. Conclusions

On the basis of the data which have been obtained it is possible to reach the following conclusions:

- 4.1 The test procedures described in par. 3.11 are capable of discriminating between MIL-L-23699 oils with total acid numbers above and below 2.0 mg.KOH/gram with an accuracy of about 0.05 mg.KOH/gram. They have been applied successfully to several used oil samples. See par. 3.12.
- 4.2 The test procedures are applicable to MIL-H-23699 oils containing either type A or type B additive packages as described in par 3.11.

- 4.3 If the additive type in the oil to be tested is not known with certainty, the following method would be followed:  
To 5 ml of the oil to be tested add 20 ml of acetone, 10 drops of phenolphthalein indicator solution and 6.3 ml of 0.0297 N aqueous sodium hydroxide solution. If any violet color is observed in the lower layer, the mixture is shaken without any further additions of reagents. If no violet color is observed, an additional 1.0 ml of 0.0297 N aqueous sodium hydroxide solution and 1.0 ml of 0.5% w 1,4-dihydroxyanthraquinone solution are added and the mixture shaken.
- 4.4 The test procedure lends itself to ready packaging as follows:
- Acetone - 20 ml vials
  - Sodium hydroxide solution (0.0297 N aqueous)
    - 6.3 and 1.0 vials
  - Phenolphthalein Indicator - dropper bottle.
  - 0.5% w 1,4-dihydroxyanthraquinone solution
    - 1.0 ml vial
- Sample - small plastic measuring cup with line at 5 ml mark.
- Alternatively, the shaking bottle may be marked with lines at 5 ml for the sample 25 ml for sample + acetone, 31.3 ml for sample + acetone + 6.3 ml sodium hydroxide solution, and 32.3 ml and 33.3 ml for additional 1.0 ml quantities of sodium hydroxide and 1,4-dihydroxyanthraquinone solution.
- 4.5 The method described in par. 4.3 for the determination of additive type appears to be reliable except in the case of extremely oxidized or hydrolyzed samples in which the 1,4-dihydroxyanthraquinone has been lost from the sample through oxidation or hydrolysis. The additional sodium hydroxide and additive added to such samples would not be likely to bias the test results for the following reasons: Although the sample would be treated as a type A additive material even though it was really a highly oxidized type B material, the high state of sample oxidation would be sufficient to prevent a "pass" reading from being obtained even though an apparently too large amount of base was added. Similar considerations apply to highly hydrolyzed samples, although in that case the situation is not as clear cut. In that case the most reliable results will be obtained when the additive type in the oil sample is known beforehand.

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